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METHOD OF TESTING COLD APPLIED TWO-COMPONENT POLYSULFIDE POLYMER TYPE JOINT SEALING COMPOUND

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH**" in Part X of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

OVERVIEW

This method describes the test procedures for the determination of the properties specified for machine extruded or pourable cold applied, two-component, polysulfide polymer type joint-sealing compounds. The base component containing polysulfide polymer shall be referred to as Component B and the component containing the catalyst or accelerator shall be referred to as Component A.

This test method is divided into the following parts:

- I. Procedure for Mixing Components
- II. Method of Testing Penetration at 25 and 70°C
- III. Method of Determining Cold Flow
- IV. Method of Determining Resilience of an Original Sample at 25 and 70°C
- V. Method of Determining Resilience of Oven-Aged Specimen
- VI. Method of Determining Bonding to Concrete

VII. Method of Determining Non-Volatile Content

VIII. Method of Determining Viscosity of Components for Machine Extruded Joint Sealants

IX. Method of Determining Viscosity and Application Time of Pourable Joint Sealants

X. Safety and Health

PART I. PROCEDURE FOR MIXING COMPONENTS

A. SCOPE

This part of the test method describes the preparation and mixing of the two components of a joint sealant. Incomplete mixing or improper proportioning of the components will not give true test results.

B. PREPARATION AND MIXING

1. See the "Precautions" on page 8.
2. Hand-mixing procedures for machine-extruded materials:

- a. Preparation. See the "Precautions" on page 8. Combine the components and stir vigorously for 30 ± 5 s using a firm tempered spatula with a 25-mm, square-tipped blade.
- b. Scrape the sides and bottom of the container occasionally during this mixing period. Mix one batch for the cold flow and volatile tests, and mix a second batch for the penetration, resilience and bond tests.
- c. While pouring the samples, do not scrape the sides or bottom of the container.

C. APPARATUS, POURABLE MATERIAL

A laboratory mixer with a propeller-type paddle, 64 mm in diameter, operating at 85 ± 10 rad/s is required.

D. PREPARATION AND MIXING

1. See Precautions.
2. Weigh a sufficient amount of the two components in the ratio given by the manufacturer to give a volume of about 500 to 1000 mL.
3. Mix the materials for 5 min in a can of approximately twice the volume of the sample, using the mixer described in Section C.

PART II. METHOD OF TESTING PENETRATION AT 25 AND 70°C

A. SCOPE

This part of the test method is essentially the same as ASTM Designation: D 217. Penetration is expressed as the distance measured to the nearest 0.1 mm that the cone penetrates vertically into a sample.

B. APPARATUS

1. A penetrometer shall be used as specified in ASTM Designation: D 217. See Precautions.
2. Seamless ointment containers shall have an approximate diameter of 95 mm and

approximate height of 32 mm. Each container shall have an approximate volume of 180 mL.

3. A cone is required as specified in ASTM Designation: D 217. The total load on the sample, including the penetration cone and penetrometer shaft, shall be 150 g.
4. A forced air circulated oven capable of maintaining a temperature of $70 \pm 1^\circ\text{C}$ is required.

C. PREPARATION AND TESTING OF SAMPLES AT 25°C

1. Fill the 180 mL containers with the compound; strike off and smooth the surface. Allow the specimens to cure 24 h or other specified time at room temperature.
2. Condition the sample and cone at $25 \pm 1^\circ\text{C}$ for 1 h.
3. Using the penetration cone attachment with the cone set at its highest position and the dial set at zero, lower the indicator assembly until the tip of the cone is in contact with the surface of the specimen. Lock in place.
4. Release the cone shaft locking mechanism for 5 s to permit the cone to penetrate the surface being tested.
5. Lock the cone shaft, push dial needle shaft down to meet the cone shaft and read the indicating dial.
6. Make three tests for penetration on each sample at points on the surface not less than 19 mm apart and 19 mm from the edge of the container.

D. PREPARATION AND TESTING OF SAMPLES AT 70°C

1. Prepare the samples and allow them to cure as described in Section C.
2. Condition the sample and cone in an oven at $70 \pm 1^\circ\text{C}$ for 90 min.

3. Remove the sample and cone from the oven; insert the cone in the penetrometer. Use the same procedure as in Section C to determine the penetration with the exception that the test must be completed within 20 sec after removing sample from oven. Return the cone and sample to the oven for 5 min and repeat the determination. Obtain three penetration values in this manner.

E. TEST REPORT

Report the penetration for each temperature as the average of three tests.

PART III. METHOD OF DETERMINING COLD FLOW

A. SCOPE

This part of the test method describes the procedure for determining the flow of joint sealing compounds at room temperature.

B. APPARATUS

1. Two channels, 25-mm wide, 38-mm deep and 610-mm long, formed with 0.024-mm thick of black iron, galvanized iron or aluminum sheet. Metal plates 38 by 25 by 13 mm are inserted near each end of the channels. The metal plates shall be 508 mm apart and held in place with "C" clamps. In order to support the channels in a vertical position from a nail or hook, 6-mm diameter holes are cut in the bottom of the channels, 13 mm from the end.

C. PREPARATION OF SAMPLES

1. Fill the channels with the sealing compound from the bottom to the top without the entrapment of air.
2. Strike off the excess compound.
3. Remove the plates from one channel at 3 min after filling.
4. Suspend the specimen vertically with the end last filled down.
5. Remove the plates from the second channel 40 min after filling.

6. Suspend the specimen vertically with the end last filled down.

D. TEST REPORT

Report the total movement of the bottom of the specimen 1 h after removing the plates, in mm.

PART IV. METHOD OF DETERMINING RESILIENCE OF THE ORIGINAL SAMPLE AT 25 AND 70°C

A. SCOPE

This part of the test method describes the procedure for determining the resilience of joint sealing compounds. It is a measure of the resistance of the compound to infiltration of granular material.

B. APPARATUS

1. Use the apparatus for penetration as specified in ASTM Designation: D 217, except that the ball penetration tool attachment is used in place of the cone.
2. The ball penetration tool is described as follows:

The diameter of the ball is 17.0 ± 0.13 mm. The total length of the shaft is approximately 70 mm. The diameter of the shaft is 5.60 ± 0.25 mm, except that the diameter of the top 17.78-mm length of the shaft shall be 3.05 ± 0.25 mm. The mass of the ball and shaft shall be 27.5 ± 0.1 g. The total load on the sample, including the ball penetration tool and penetrometer shaft, shall be 75 g.

3. An oven shall be used as specified in Part II, Section B.

C. PREPARATION AND TESTING OF SAMPLES AT 25°C

1. Prepare the samples and condition them in the same manner as for the penetration test at 25°C. See Part II, Section C.

2. Using the ball penetration tool attachment with the ball set at its highest position and the dial set at zero, lower the indicator assembly until the ball is in contact with the surface of the specimen. Lock in place.
3. Release the locking mechanism, then apply a uniform pressure to the ball, by manual loading, so that the ball penetrates the specimen to a dial reading of 100 at a uniform rate in 10 sec.
4. Lock the ball shaft locking mechanism for 5 sec. During this time, raise the dial shaft to its zero position. The needle should read zero.
5. Release the ball shaft locking mechanism for 20 sec at the end of the above prescribed 5 sec. Lock the shaft, push dial needle shaft down to meet the ball and read the indicating dial.
6. Observing the same spacing between tests as for penetration (see Part II, Section C), make two additional tests.

D. PREPARATION AND TESTING OF SAMPLES AT 70°C

1. Prepare and cure the samples using the procedure to determine penetration. See Part II, Section C.
2. Condition the samples and ball penetration tool for 90 min in an oven at $70 \pm 1^\circ\text{C}$.
3. Remove the penetration tool and sample from the oven. Insert the tool in the penetrometer and complete the resilience reading within 55 sec after removing the sample from the oven.
4. Return the sample and tool to the oven for 10 min before making another reading.
5. Obtain three readings in the above manner.

E. TEST REPORT

Report for each temperature, the average of three resilience values, each calculated as 100 minus the dial reading 20 sec after release.

PART V. METHOD OF DETERMINING RESILIENCE OF OVEN-AGE SPECIMEN

A. SCOPE

The resilience of an oven-aged sample of joint sealing compound is intended to measure its resistance to infiltration of mineral matter after the compound has been subjected to an elevated temperature.

B. APPARATUS

The apparatus used is the same as described in Part IV.

C. PREPARATION AND TESTING OF SAMPLES

1. Place the specimen from the resilience test, of the original sample used in Part IV, in a forced air circulated oven maintained at $70 \pm 1^\circ\text{C}$.
2. After seven days, remove it from the oven and condition in air for 2 h at $25 \pm 1^\circ\text{C}$.
3. Then make three tests for resilience as described in Section C of Part IV.

D. TEST REPORT

Report the average of three resilience values of the oven-aged samples, in %. Each calculated value is 100 minus the dial reading 20 sec after the release.

PART VI. METHOD OF DETERMINING BONDING OF CONCRETE

A. SCOPE

This part of the test method is intended to measure the adhesion of joint sealing compounds to clean dry or moist portland

concrete cement with sawed faces when subjected to a temperature of -29°C .

B. APPARATUS

1. Bond extension machine: The apparatus shall be capable of extending the bond specimens as described below from a spacing of 12.7 mm between blocks to 25.4 mm at a uniform rate of 3.2 mm/h at -29°C . ASTM Designation: C 719 shows a machine which meets this requirement; however, machines of other designs may also be satisfactory.
2. Cooling chamber: A refrigerated enclosure shall maintain the specimens mounted in the bond extension machine at $-29^{\circ}\text{C} \pm 1^{\circ}\text{C}$.
3. Brass spacers are required for molding the specimens: 12.7 by 12.7 by 50.8 mm.
4. Tin-plated panels shall consist of the following: 76.2 by 127 mm.
5. Molds for concrete blocks: The mold shall be of metal and shall be provided with a detachable metal base plate. Means shall be provided for securing the base plate to the mold. The assembled mold and base plate shall be watertight and shall be oiled with mineral oil before use. The inside measurement of the mold shall be 255-mm wide by 445-mm long by 76.2-mm high.
6. A concrete saw having a diamond or silicon carbide cutting edge is required. Blades to be of such size that the saw cut does not exceed 9.5 mm in width.
7. A drying oven is required. It shall regulated at $104 \pm 3^{\circ}\text{C}$.

C. MATERIALS USED IN CONCRETE BLOCKS

1. Aggregate: The aggregate shall conform to the requirements in Section 90 of Caltrans Standard Specifications and the following detailed requirements:

The coarse aggregate shall consist of crushed limestone, have an absorption of not more than 1.5 %, as determined by California Test 206, and shall conform to the following grading:

Sieve Size	Percent Passing
19-mm	98.5 ± 1.5
12.5-mm	66 ± 3
9.5-mm	33 ± 3
4.75-mm	1.5 ± 1.5

The fine aggregate shall consist of natural siliceous sand and shall conform to the following grading:

Sieve Size	Percent Passing
4.75-mm	100
2.36-mm	85 ± 3
1.18-mm	65 ± 5
600- μm	45 ± 5
300- μm	21 ± 5
150- μm	7 ± 2

2. Portland Cement: The portland cement shall conform to the requirements in Section 90 in Caltrans Standard Specifications.

D. DESIGN OF CONCRETE MIX

1. The concrete shall have an approximate water-cement ratio of 0.48 that produces concrete with a slump of 65 ± 15 mm.
2. The ratio of fine aggregate to total aggregate shall be approximately 40 %, by solid volume.
3. The cement content shall be 350 kg/m³ of concrete.
4. The air content shall be 5.0 ± 0.5 % and shall be obtained by the addition to the batch as mixed of a sufficient quantity of an air-entraining admixture.

E. FABRICATION OF TEST BLOCKS

1. Mix the concrete, fill the mold and moist cure the block in accordance with ASTM Designation: C 192.

2. After moist curing the specimens for not less than 28 days, saw the concrete block into 25 by 51 by 76-mm test blocks in the following manner:
 - a. First cut the 254 by 445 by 76-mm block in half by making a cut down the long axis through the center. This yields two slabs.
 - b. Cut each into two 58 by 444 by 76-mm slabs by making cuts at a distance of 58-mm from the sawed faces.
 - c. Then saw each of the four 58 by 444 by 76-mm slabs obtained as described above, into twelve 25 by 51 by 76-mm blocks in the following manner:
 - d. Cut a 13-mm portion from the 51 by 76-mm face which was in contact with the mold face. Then cut twelve 25 by 51 by 76-mm blocks from the slab.
 - e. Each 254 by 444 by 76-mm slab yields forty-eight 25 by 51 by 76-mm test blocks. These dimensions permit sawing by blades up to 9.5-mm wide.
 3. After sawing and while still wet from the sawing operations, lightly scrub the surfaces of the blocks with a stiff-bristled brush while holding under a stream of running water.
 4. Then store the blocks in the laboratory air in such a manner as to preclude dust accumulation on the surfaces.
- stiff-bristled brush and place the blocks in a desiccator until used.
3. Soak six blocks in water for 24 h. Remove the blocks from water and drain off the excess moisture before assembling then for testing, as described below.
 4. Prepare each of the three dry-bond specimens by placing two dry concrete blocks on a base plate with the 25 by 76-mm faces in contact with the base plate.
 5. Space the concrete blocks 13 mm apart by means of blocks 13 by 13 by 51-mm standing on their 13 by 13-mm ends and forming an opening between them 13 by 51-mm long. Use clamps, tape or other suitable means to hold the blocks in position.
 6. Prepare the six wet-bond blocks using the same procedure as with the dry-bond blocks. Put the wet-bond blocks into water until immediately before filling the spaces between them.
 7. Fill the spaces between the dry-bond blocks with the freshly mixed compound to be tested from the bottom to the top to exclude air pockets. Strike off excess overfill with a spatula.
 8. Immediately before filling each wet-bond specimen, remove from water and drain. Wipe off excess moisture from all surfaces of the hole to be filled, using a dry absorbent swab. Fill in the same manner as for the dry-bond specimens.

F. PREPARATION OF TEST SPECIMENS

1. Prepare six test specimens - three for the dry-bond and three for the wet-bond tests. Two blocks are required for each specimen.
2. Dry six blocks for the dry-bond test to a constant weight in an oven maintained at $104 \pm 3^{\circ}\text{C}$. Then brush the blocks with a

G. TESTING THE SPECIMENS

1. After conditioning the specimen blocks for 24 h at room temperature, place them in the holders of the bond extension machine for further conditioning at $-29 \pm 1^{\circ}\text{C}$ for 1 h.
2. Place slight tension on the specimens.

3. Start the bond extension machine.
4. Operate the machine for the time required to obtain the specified extension. (2 h for 50 % or 4 h for 100 % extension).
5. Stop the bond extension machine and release the tension on the concrete block specimens.
6. Remove the specimens from the machine, expose them to room temperature for 30 min, then examine them immediately for defects described in Section H.
7. Repeat the above procedure twice which comprises three cycles or one complete test.

H. INTERPRETATION OF TEST RESULTS

A bond block shall be considered a failure if a separation in the sealing compound or a separation between the sealing compound and concrete block occurs which, when measured perpendicularly to the face of the sealant, is in excess of the following amount:

- | | |
|--------------------|------|
| a. Dry-bond blocks | 3 mm |
| b. Wet-bond blocks | 6 mm |

PART VII. METHOD OF DETERMINING NON-VOLATILE CONTENT

A. SCOPE

This part of the test describes the procedure for determining the non-volatile content of joint sealant at 70°C.

B. PREPARATION AND TESTING OF SAMPLE

1. Transfer 3 to 5 g of freshly mixed material to a tared dish, approximately 64 mm in diameter. Spread the material with a 25.4-mm wide square ended spatula to cover the bottom of the dish with a uniform thickness of about 0.79 mm.
2. Weigh to the nearest 1 mg and immediately place the sample in a forced air circulated oven for 24 h at $70 \pm 1^\circ\text{C}$.

3. Cool the sample to room temperature and reweigh to nearest 1 mg.
4. Calculate the % total non-volatile as follows:

$$\% \text{ non-volatile} = (\text{Final mass of the sample} \times 100) / \text{Initial mass of the sample}$$

PART VIII. METHOD OF DETERMINING VISCOSITY OF COMPONENTS FOR MACHINE EXTRUDED JOINT SEALANTS

A. SCOPE

This part of the test describes the procedure for determining the original viscosity of the two components used to make machine extruded joint sealants.

B. APPARATUS

Brookfield viscometer: This device shall be capable of operating at 5.2 rad/s. It shall be equipped with a No. 5 spindle.

C. PREPARING AND TESTING SAMPLES

1. Mix each component until it is uniform. See Precautions.
2. Fill a 500-mL or 1-L can approximately 75 % full with the component to be tested. Adjust the temperature of the sample to 25°C.
3. Determine the viscosity using the No. 5 spindle on the Brookfield viscometer set at 5.2 rad/s.

PART IX. METHOD OF DETERMINING VISCOSITY AND APPLICATION TIME OF POURABLE JOINT SEALANTS

A. SCOPE

This part of the test describes the procedure for determining the viscosity of pourable joint sealants 5 min after mixing and also for determining the application time or pot life.

B. APPARATUS

1. In addition to the apparatus described in Part VIII, use a No. 6 spindle for the viscometer.

C. PREPARING AND TESTING SAMPLES

1. Adjust the two components to 25°C.
2. Mix the sample as described in Part I of this test method.
3. Fill a 500-mL or 1-L can approximately 75 % full.
4. When 5 min has elapsed after the end of the mixing period, determine the viscosity using the No. 5 spindle at 5.2 rad/s.
5. Remove the No. 5 spindle from the viscometer. Clean the spindle at once. Replace it with a No. 6 spindle. Adjust the mixture to 25°C.
6. At 5-min intervals, determine the viscosity using the No. 6 spindle at 5.2 rad/s.

D. TEST REPORT

Report application time as the time at which the viscosity of the sealant is first equal to or greater than that specified for application time.

E. PRECAUTIONS TO BE NOTED IN PERFORMING TESTS

1. Mixing of Individual Components Before Testing:

It is important that each individual component be uniformly mixed by boxing and vigorous stirring before any tests are performed. This is especially important with Component A if it appears in liquid form. In this case, extreme care must be

taken to insure that all settled material is uniformly reincorporated.

2. Penetrometer:

Keep penetrometer in good working order. Avoid conditions such as a bent plunger or dirt or corrosion in the guide bearings which might cause binding or other irregularities in the movement of the plunger.

- a. Care of the Cone:

Handle the cone carefully to avoid bending of the shaft or dulling of the point. If the cone is cleaned with a solvent, be sure the solvent has evaporated completely before use of the cone.

PART X. SAFETY AND HEALTH

This method may involve hazardous materials, operations and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of whomever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Observe good hygiene practices. Wash hands after handling samples and before eating, drinking or smoking.

Prior to handling, testing or disposing of any of materials, testers are required to read applicable sections of the Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

REFERENCES:

California Test 206
ASTM Designations: C 192, C 719, and D 217
Caltrans Standard Specifications

End of Text (California Test 413 contains 8 pages)